UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

DATE: 08-MAY-2001

SUBJECT: PP# 1F03935. Triclopyr in Fish and Shellfish. **Evaluation of Residue Data and Analytical Methods**. MRID#s 440151-01, 444561-02 thru -04, 444561-08, 444561-12, and 451709-01. Barcode D268064. Chemical 116001. Case 281961. Submission S583618.

FROM: William H. Donovan, Ph.D., Chemist
Registration Action Branch 1 (RAB1)
Health Effects Division (HED) (7509C)

THRU: G. Jeffrey Herndon, Branch Senior Scientist
RAB1/HED (7509C)

TO: Jim Tompkins/James Stone, PM Team 25
Registration Division (7505C)

DowElanco has submitted a petition to establish permanent tolerances for residues of the herbicide triclopyr (Garlon® 3A) in fish and shellfish. The herbicide is intended to control aquatic weeds growing in lakes, ponds, reservoirs, and wetlands, and to control woody brush and herbaceous weeds in wetlands and on the banks and shores of aquatic sites. Tolerances are proposed for the combined residues of triclopyr, (3,5,6-trichloro-2-pyridinyl)oxyacetic acid, and its metabolites 3,5,6-trichloro-2-pyridinol (TCP) and 2-methoxy-3,5,6-trichloropyridine (TMP) in or on the following raw agricultural commodities:

Fish	3.0 ppm
Shellfish	5.0 ppm

It is further proposed that an allowable water residue level of 0.5 ppm be established for triclopyr *per se* in compliance with the Safe Drinking Water Act. However, HED does not regulate pesticide residues in potable water.

Currently, tolerances are established for the combined residues of triclopyr, TCP, and TMP in or on grass forage and grass hay [40 CFR §180.417 (a)(1)], and for residues of triclopyr and TCP in or on eggs; the meat, fat, and meat byproducts (except liver and kidney) of cattle goats, hogs,

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horses, and sheep; the meat, fat, and meat byproducts (except kidney) of poultry; milk; liver and kidney of cattle goats, hogs, horses, and sheep; rice grain; and rice straw [40 CFR §180.417 (a)(2)]. Additionally, time-limited tolerances with an expiration/revocation date of 6/30/2000 for the combined residues of triclopyr, and its metabolite TCP in fish and shellfish were listed under 40 CFR §180.417 (b).

The attached contractor's document has been reviewed and revised to reflect current HED policy.

Executive Summary of Chemistry Deficiencies

- Agency validation of the analytical method for fish.
- Radiovalidation of the analytical method for fish.
- Revised Section F.

RECOMMENDATIONS

Provided that the petitioner submits a revised Section F, an adequate radiovalidation study, and that agency validation of the analytical method is successful, HED concludes there are no residue chemistry data requirements that would preclude establishment of the proposed tolerances for triclopyr and its TCP and TMP metabolites in/on fish and shellfish. A human-health risk assessment will be prepared as a separate document.

List of Attachments

Attachment 1. Contractor review.
Attachment 2. IRLS sheet for triclopyr.

cc (with Attachments): W. Donovan
RDI: RAB1 Chemists (20-MAR-2001), G. Herndon (08-MAY-2001)
W.H. Donovan:806R:CM#2:(703)305-7330:7509C:RAB1

ATTACHMENT 1

Triclopyr
PC Code 116001
(DP Barcode D268064)

PP#1F03935: Evaluation of Residue Chemistry Data to Support Permanent Tolerances for Residues in Fish and Shellfish

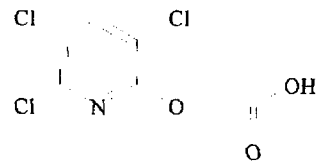
December 15, 2000

Contract No. 68-W-99-053

Submitted to:
U.S. Environmental Protection Agency
Arlington, VA

Submitted by:
Dynamac Corporation
1910 Sedwick Road
Durham, NC 27713

TRICLOPYR



PP#1F03935: Evaluation of Residue Chemistry Data to Support Permanent Tolerances for Residues in Fish and Shellfish

INTRODUCTION

DowElanco has submitted a petition to establish permanent tolerances for residues of the herbicide triclopyr (Garlon® 3A) in fish and shellfish. The herbicide is intended to control aquatic weeds growing in lakes, ponds, reservoirs, and wetlands, and to control woody brush and herbaceous weeds in wetlands and on the banks and shores of aquatic sites. Tolerances are proposed for the combined residues of triclopyr, (3,5,6-trichloro-2-pyridinyl)oxyacetic acid, and its metabolites 3,5,6-trichloro-2-pyridinol (TCP) and 2-methoxy-3,5,6-trichloropyridine (TMP) in or on the following raw agricultural commodities:

Fish	3.0 ppm
Shellfish	5.0 ppm

It is further proposed that an allowable water residue level of 0.5 ppm be established for triclopyr *per se* in compliance with the Safe Drinking Water Act. However, HED does not regulate pesticide residues in potable water.

Currently, tolerances are established for the combined residues of triclopyr, TCP, and TMP in or on grass forage and grass hay [40 CFR §180.417 (a)(1)], and for residues of triclopyr and TCP in or on eggs; the meat, fat, and meat byproducts (except liver and kidney) of cattle goats, hogs, horses, and sheep; the meat, fat, and meat byproducts (except kidney) of poultry; milk; liver and kidney of cattle goats, hogs, horses, and sheep; rice grain; and rice straw [40 CFR §180.417 (a)(2)]. Additionally, time-limited tolerances with an expiration/revocation date of 6/30/2000 for the combined residues of triclopyr, and its metabolite TCP in/on fish and shellfish were listed under 40 CFR §180.417 (b). However, in a meeting held on 15-JUL-1996, the HED Metabolism Committee concluded that available metabolism data supported regulation of the following: parent triclopyr only in plants, milk, poultry, and eggs; and triclopyr and TCP in meat and meat byproducts (Memo, W.O. Smith, 23-JUL-1996).

Seven volumes of chemistry data are evaluated in this document.

CONCLUSIONS

OPPTS 830 Series GLNs: Product Properties

1. Product chemistry data were reviewed in conjunction with the Triclopyr Reregistration Eligibility Decision dated 27-OCT-1998. No additional product chemistry data are required for this petition.

OPPTS GLN 860.1200: Proposed Uses

2. The proposed aquatic site use directions for the 3 lb/gal SC/L formulation of the triethylamine salt of triclopyr (Garlon® 3A, EPA Reg No. 62719-37) are adequate. The restrictions are adequate to ensure that triclopyr residues in potable water will not exceed 0.5 ppm and that residues in irrigation water will be nondetectable. However, establishing a 0.5 ppm residue limit for water is not under the purview of HED.

OPPTS GLN 860.1300: Nature of the Residue in Plants and Livestock

3. Adequate plant and livestock metabolism data were submitted in support of the currently established tolerances, and are summarized in the Triclopyr Reregistration Eligibility Decision (27-OCT-1998). No additional plant or livestock metabolism data are required for this petition. The HED Metabolism Assessment Review Committee (MARC) determined that, in light of new TCP toxicology data, no changes to the existing tolerance expressions for plants and livestock commodities are warranted (D274243, W. Donovan and W. Dykstra, 27-APR-2001).

OPPTS GLN 860.1340: Residue Analytical Method - Water, Fish, and Shellfish

4a. GC/MS method GRM 95.18 is adequate for collection of triclopyr, TCP, and TMP residue data from water. The stated LOQ is 0.1 ppb (0.0001 ppm) for each analyte. Triclopyr and TCP residues in water were also analyzed using an immunoassay method GRM 95.11. The LOQs were 0.145 ppb for triclopyr and 0.516 ppb for TCP. The immunoassay method is adequate for analysis of triclopyr and TCP in water.

4b. GC/MS method GRM 97.02 is adequate for collection of triclopyr residue data from fish and shellfish tissue. The nominal LOQs for triclopyr, TCP, and TMP are 0.01 ppm for each analyte. LOQs calculated as 10x the standard deviation of recoveries of 0.01 ppm fortifications were up to 0.023 ppm.

4c. Method GRM 97.02 has undergone a successful independent laboratory validation (ILV) and has been forwarded to EPA's Analytical Chemistry Branch (ACB) for a Petition Method Validation (PMV) (D264679, W. Donovan, 07-APR-2000). The petitioner should submit the results of a radiovalidation study to ensure the ability of the analytical method to recover aged residues. **Until successful method validation of the analytical**

method is reported by ACB, the data requirement for analytical methods has not been satisfied.

OPPTS GLN 860.1360: Multiresidue Method

5. The FDA PESTDATA database dated 1/94 (PAM Vol. I, Appendix I) indicates that triclopyr is completely recovered (<80%) using the multiresidue method given in PAM Vol. 1 Section 402. Data pertaining to multiresidue methods testing of triclopyr and its metabolites through Protocols B, C, D, and E have been submitted and forwarded to FDA (Triclopyr RED, 27-OCT-1998).

OPPTS GLN 860.1380: Storage Stability Data

6. A storage study with fish and shellfish was included with this petition. Triclopyr, TCP, and TMP were stable for up to 19 months in crayfish and 24 months in bluegill. The maximum sampling-to-analysis interval for fish and shellfish residue samples was 21 months; thus, the storage stability data cover the sample storage intervals in the metabolism and magnitude of the residue (MOR) studies.

OPPTS GLN 860.1400: Water, Fish, Shellfish

Fish metabolism

7a. Bluegill sunfish were exposed to pyridine-labeled [^{14}C]triclopyr at a nominal concentration of 7.5 ppm (3x the proposed rate) for 10 days. The total radioactive residues (TRR) were 0.515 and 0.369 ppm in edible fillet muscle after 7 and 10 days of exposure, respectively [10 and 6% of the radioactivity in whole fish (5.018 and 6.029 ppm)]. Residues did not concentrate in fish relative to the concentration in water of 7.1-7.7 ppm. Of the TRR, 74-76% were characterized. According to this study, the major residues of triclopyr in fish are the parent compound and TCP and their conjugates.

7b. HED notes that the metabolic profile of triclopyr in fish from this metabolism study is not consistent with the results reported in the MOR studies. Specifically, this study found TMP to comprise <1% TRR, while in the MOR studies TMP was consistently found to be the primary residue in bluegill fillet samples after exposure to triclopyr-treated water for 1 day or more. The MOR studies analyzed edible fish tissues using an analytical method that successfully passed ILV and was demonstrated to successfully recover TMP residues from fish samples (80-101% recovery over a fortification range of 0.010 - 5.00 ppm). In contrast, the fish metabolism study analyzed whole fish samples using a different extraction procedure that was not subjected to ILV and gave a mean extraction efficiency of TMP-fortified fish samples of just 60 ± 10% over an unspecified range. Thus, HED finds that the weight-of-the-evidence favors the results of the MOR studies that suggest the residues of concern in triclopyr-treated fish are the parent, TCP, and TMP.

7c. The HED MARC determined that the residues of concern in fish and shellfish are triclopyr, TCP, and TMP (D274243, W. Donovan and W. Dykstra, 27-APR-2001). The Committee also determined that the residues of concern in drinking water are triclopyr and TCP.

Magnitude of the residue

8a. In a study on triclopyr in two locations in Lake Minnetonka, MN (1994), water was treated with triclopyr at a nominal concentration of 2.5 ppm. Water and fish were sampled at intervals from time zero through 4 weeks posttreatment.

8b. Maximum combined residues in edible fish tissues were 0.952 ppm in bass, 0.754 ppm in bluegill, 0.275 ppm in bullhead, and 0.678 ppm in sucker. The metabolite TMP accounted for most of the combined residues, with only minor amounts of triclopyr and TCP. Maximum residue levels were attained at 1 day to 1 week posttreatment. At the end of 4 weeks, residues in fish decreased to <0.06 ppm.

8c. In shellfish, maximum combined residues were 0.523 ppm in edible clam tissue and 0.454 ppm in crayfish, 3 days after treatment. After 4 weeks, residues were ≤0.07 ppm. At early time points, triclopyr was the predominant compound, then after 3 days most of the combined residue in clams consisted of TMP, and the three analytes were detected at comparable levels in crayfish.

8d. The maximum total residue of triclopyr, TCP, and TMP in water from the treated Lake Minnetonka plots were 3.73 ppm, 3.70 ppm of which was parent, and were reached at 3 hours after treatment. After 5 days, combined residues were 0.7 and 1.8 ppm; residues fell below 0.5 ppm after 1-2 weeks. The maximum off-plot combined total residue level was 0.294 ppm 100 m from the treated plot. Throughout the study, TCP and TMP residue levels were very low compared to parent.

9a. In concurrent 1995 studies in CA, MO, and TX, ponds were treated with triclopyr to a nominal concentration of 2.5 ppm. Water and fish were sampled from time zero through 12 and 4 weeks, respectively, and analyzed for combined residues of triclopyr, TCP, and TMP.

9b. Maximum combined residues were 0.956 ppm in bluegill fillet and 2.97 ppm in catfish reached after 0.5 days to 2 weeks. Data on fish viscera showed decreases in triclopyr over time with concurrent increases in metabolite levels, whereas, in fillet tissues, TMP levels were substantially higher than those of the other analytes.

9c. Total triclopyr, TCP, and TMP residues in water reached maximum levels of 2.1-2.8 ppm at 1-3 hours posttreatment in CA, 1 day in MO, and 6 hours in TX. Total residues decreased to levels below 0.5 ppm after 3 weeks in all sites with the exception of one CA site, in which the 3-week level was 0.645 ppm with residues falling below 0.5 ppm after 4 weeks. Throughout the tests, little degradation of triclopyr in water was observed;

maximum TCP levels were 0.004-0.022 ppm and maximum TMP concentration was 0.004-0.007 ppm.

10a. In a 1996 study in TX, pond water was treated with triclopyr to a nominal concentration of 2.5 ppm. Water and fish were sampled from time zero through 6 weeks and analyzed for combined residues of triclopyr, TCP, and TMP.

10b. Maximum residues in green sunfish and catfish fillets were 0.723 and 2.04 ppm, respectively, after 3 and 7 days, and consisted almost entirely of TMP.

10c. Maximum total residues in pond water were 2.44 ppm, consisting primarily of the parent compound; TCP and TMP concentrations were very low, ≤0.013 and ≤0.002 ppm, respectively. Total residues in water decreased to below 0.5 ppm on Day 13 after treatment.

11. Current HED policy recommends regulating residues found in the *edible portions* of fish and shellfish. In the submitted studies, the maximum combined residue in fish edible portions was 2.97 ppm. The maximum combined residues in shellfish edible portions were 0.523 ppm in clams and 0.454 ppm in crayfish. In a study previously reviewed ("91-MN-06. Proposed Section 18 exemption for the use of triclopyr in or near aquatic sites", D. McNeilly, 03-APR-1991), maximum combined residues were 3.44 ppm in clams (edible portion) and 4.87 ppm in crayfish (whole organism including shell) from Lake Seminole, GA treated to a concentration of 2.5 ppm triclopyr in the water. Because the crayfish were analyzed as whole organisms, it is not appropriate to use these data to set tolerance levels. Consequently, the appropriate tolerance levels for combined residues of triclopyr, TCP, and TMP in fish and shellfish are 3.0 and 3.5 ppm, respectively. **A revised Section F specifying these tolerance levels should be submitted.**

OPPTS GLN 860.1480: Meat, Milk, Poultry, Eggs.

12. The proposed tolerances would not alter the maximum theoretical dietary burdens (MTDBs) of triclopyr for cattle or poultry estimated in conjunction with the existing feeding studies for triclopyr use in/on grass and rice. Additional feeding study data are not required.

OPPTS GLN 860.1850/1900: Confined/Field Accumulation in Rotational Crops

13. There are no rotational crop issues associated with the proposed use.

Codex Issues

14. There are no established or proposed Codex, Canadian, or Mexican MRLs for triclopyr residues. Therefore, harmonization is not an issue at this time.

RECOMMENDATIONS

Provided that the petitioner submits a revised Section F, an adequate radiovalidation study, and that agency validation of the analytical method is successful, HED concludes there are no residue chemistry data requirements that would preclude establishment of the proposed tolerances for triclopyr and its TCP and TMP metabolites in/on fish and shellfish. A human-health risk assessment will be prepared as a separate document.

DETAILED CONSIDERATIONS

OPPTS 830 Series GLNs: Product Properties

Product chemistry data were reviewed in conjunction with the Triclopyr Reregistration Eligibility Decision (RED) dated 27-OCT-1998. No additional product chemistry data are required for this petition.

OPPTS GLN 860.1200: Directions for Use

Garlon® 3A, the 3 lb/gal SC/L formulation of triclopyr triethanolamine salt, is proposed for use on aquatic sites to control submerged, floating, and emerged weeds, including woody plants, in lakes, ponds, reservoirs, and marshes, including banks and shores, and in non-irrigation ditches or canals with no continuous outflow.

For control of floating and emerged weeds, triclopyr is proposed for foliar application at 1.5-6 lb ae/A. Application may be repeated as necessary, not to exceed 6 lb ae/A/season. For ponds, lakes, and reservoirs, surface applications are made in 20-200 gal/A and aerial applications are made in a minimum of 10 gal/A.

To control submerged weeds, application rates are to be selected to provide a triclopyr concentration in treated water of 0.75-2.5 ppm ae, according to a table showing the quantity of triclopyr needed to achieve target concentrations in water depths from 1 - 10 feet. Retreatment is permitted, although the total seasonal application may not exceed 2.5 ppm.

To control woody and broadleaf plants in wetland areas, including areas adjacent or surrounding domestic water supply reservoirs, lakes, and ponds, triclopyr is applied at 6-9 lb ae/A to woody plants and at 1-3 lb ae/A to broadleaf weeds.

Applications may be made using ground (or water surface) equipment using 20-200 gal/A or using aerial equipment in 10-30 gal/A.

Restrictions:

1. Irrigation. Do not use treated water for irrigation for 120 days after application or until laboratory analysis by immunoassay indicates nondetectable triclopyr concentration.
2. Lakes and reservoirs. Application is restricted to minimum set-back distances from functioning potable water intakes. For surface weed treatment, the set-back distances range from 500-1300 feet for ≤4 to >16 acres treated at the maximum application rate of 2 gallons/A. For submerged weed treatment, the set-back distances range from 1000-2000 feet for ≤4 to >16 acres treated at the maximum application rate needed to achieve a triclopyr water concentration of 2.5 ppm (e.g., 2.3 gallons/A in a water depth of 1 foot). If application is closer than the specified setback, potable water intakes must be turned off for 5 days following application or until triclopyr level is <0.5 ppm, as determined by laboratory analysis or immunoassay.
3. Ponds. Functioning potable water intakes must be turned off for 21 days or until triclopyr level is <0.5 ppm, as determined by laboratory analysis or immunoassay.

Conclusions: The proposed aquatic site use directions for the 3 lb/gal SC/L formulation of the triethylamine salt of triclopyr (Garlon® 3A, EPA Reg No. 62719-37) are adequate. The restrictions are adequate to ensure that triclopyr residues in potable water will not exceed 0.5 ppm and that residues in irrigation water will be nondetectable. However, establishing a 0.5 ppm residue limit for water is not under the purview of HED.

OPPTS GLN 860.1300: Nature of the Residue in Plants and Livestock

Adequate plant and livestock metabolism data were submitted in support of the currently established tolerances, and are summarized in the Triclopyr RED. No additional plant or livestock metabolism data are required for this petition. The HED MARC determined that, in light of new TCP toxicology data, no changes to the existing tolerance expressions for plants and livestock commodities are warranted (D274243, W. Donovan and W. Dykstra, 27-APR-2001). Fish metabolism is discussed under GLN 860.1400.

OPPTS GLN 860.1340: Residue Analytical Method - Fish, Shellfish, Water

Analysis of residues in fish. The petitioner submitted analytical method GRM 97.02 for tolerance enforcement and data collection regarding residues of triclopyr and its metabolites TCP and TMP in fish tissues. The method and validation data, which were used to generate the residue data submitted in this petition in support of the proposed tolerances, are presented in the following submission:

> 44456108 Olberding, E.L. (1997) Validation Report for the determination of Residues of Triclopyr, 3,5,6-Trichloro-2-pyridinol, and 2-Methoxy-3,5,6-trichloropyridine in Fish tissues by Capillary Gas Chromatography with Mass Selective Detection. Laboratory Study ID: RES94084. Unpublished study submitted by DowElanco. 68 p.

Using method GRM 97-02, residues are extracted from fish using 0.25 N sodium hydroxide. The extract is acidified and residues were partitioned to ethyl ether. The ether extract was passed through an alumina solid-phase extraction (SPE) column, from which TMP residues eluted with the ether and triclopyr and TCP were subsequently eluted with 0.1 N NaOH. The TMP residues were concentrated and partitioned to 1-chlorobutane. The triclopyr and TCP residues were cleaned up on C_{18} SPE prior to partitioning into 1-chlorobutane. Fluroxypyr and the fluroxypyr metabolite analogs 4-amino-3,5-dichloro-6-fluoro-2-pyridinol and 4-amino-3,5-dichloro-6-fluoro-2-methoxypyridine were added as internal standards. Samples were derivatized with *N*-methyl-*N*-(*tert*-butyldimethylsilyl)-trifluoroacetamide (MBTSTFA) to form *tert*-butyldimethylsilyl (TBDMS) derivatives of triclopyr and TCP. Samples are analyzed by capillary GC/MS. Major fragmentation ions measured were *m/z* 312, 254, 256, and 314 for triclopyr-TBDMS; *m/z* 254 and 256 for TCP-TBDMS; and *m/z* 210, 182, 211, 212, and 213 for TMP.

For method validation, untreated bluegill and catfish tissues from the aquatic dissipation and residue trials and crayfish obtained commercially were fortified with triclopyr, TCP, and TMP at 0.010, 0.050, and 0.500 ppm. The recovery of residues from fortified samples is presented in Table 1. Validation recoveries were adequate, 82-105% from samples fortified at 0.01 ppm and 77-110% overall. Concurrent recoveries obtained in conjunction with analysis of field trial samples are presented in Tables 2-4.

Limits of detection (LOD) and limits of quantitation (LOQ) were calculated as 3x and 10x standard deviation of recoveries at the target LOQ of 0.01 ppm. In this validation study, LOD ranged from 0.002-0.003 ppm and LOQs ranged from 0.007-0.009 ppm. LOQs calculated from concurrent recoveries in the residue studies ranged from 0.01 ppm to 0.023 ppm. The minimum LOQ and LOD used for residue reporting were 0.01 and 0.003 ppm, respectively.

Table 1. Validation of GC/MSD method GRM 97.02 for triclopyr, TCP, and TMP in fish and crayfish (MRID 44456108).

Sample	Fortification(ppm)	Recovery (%)		
		Triclopyr	TCP	TMP
Bluegill, edible	0.010	94, 97	92, 93	86, 89
	0.050	91	89	87
	0.500	97	81	80
	5.00	91	87	82
Bluegill, inedible	0.010	77, 92	115, 115	89, 95
	0.050	93	93	80
	0.500	90	84	99
	5.00	92	92	101
Catfish, edible	0.010	95, 97	94, 92	101, 98
	0.050	104	86	98
	0.500	92	76	92
	5.00	85	85	92

Sample	Fortification(ppm)	Recovery (%)		
		Triclopyr	TCP	TMP
Catfish, inedible	0.010	90, 95	100, 102	91, 91
	0.050	195	90	110
	0.500	86	82	96
	5.00	91	92	99
Crayfish, edible	0.010	104, 95	104, 102	82, 86
	0.050	93	100	86
	0.500	87	95	77
	5.00	86	98	81
Crayfish, inedible	0.010	102, 103	91, 92	98, 105
	0.050	98	82	92
	0.500	89	77	86
	5.00	93	84	94

Table 2. Recovery of triclopyr, TCP, and TMP (MRID 44456102) from fortified edible **fish and shellfish** samples (CA. MO, TX) using GC/MS method GRM 97.02.

Matrix	Fortification (ppm)	Number of Samples	Recovery (%) [a]		
			Triclopyr	TCP	TMP
Bass	0.010-0.011	4	57-100 (3)	89-119	71-84
	0.023-0.026	1	60	90	81
	0.050	1	74	84	85
	0.250-0.262	2	79-86	88-90	82-93
	0.509-0.525	1	85	88	73
	1.00-1.05	2	70-81	83-90	63-84
			Ave = 74	Ave = 94	Ave = 80
Bluegill	0.010-0.011	4	62-100 (3)	89-126 (1)	78-93
	0.026	1	78	97	97
	0.050	1	92	103	76
	0.250-0.262	2	88-91	88-101	78
	0.508-0.525	1	99	93	72
	1.00-1.05	2	91-97	102	65-79 (1)
			Ave = 85	Ave = 100	Ave = 81
Bullhead	0.01-0.011	4	82-100	91-98	57-110 (1)
	0.026	1	78	94	71

continued, footnotes follow

Matrix	Fortification (ppm)	Number of Samples	Recovery (%) [a]		
			Triclopyr	TCP	TMP
	0.050	1	79	106	103
	0.250-0.262	2	79-89	92-102	71-93
	0.509-0.525	1	93	90	71
	1.00-1.05	2	81-92	98-110	70-92
			Ave = 86	Ave = 98	Ave = 81
Sucker	0.010-0.011	4	76-96	87-101	75-89
	0.025-0.026	1	78	92	NA
	0.050	1	70	74	88
	0.250-0.262	2	74-87	84-87	71-98
	1.00-1.02	2	69-90	82-87	101
			Ave = 79	Ave = 90	Ave = 87
Crayfish	0.01-0.011	4	81-87	86-100	72-74
	0.026	1	82	89	87
	0.050	2	100-101	93-95	72-77
	0.250-262	2	97-105	93-99	76-89
	0.509-0.525	1	99	68	95
	1.01-1.05	1	97	94	83
			Ave = 92	Ave = 92	Ave = 79
Clam	0.010-0.011	7	88-123 (1)	88-107	69-101 (1)
	0.026	1	97	94	88
	0.050-0.052	2	80-100	65-99 (1)	81-85
	0.101-0.105	1	96	99	78
	0.250-0.262	2	85-93	73-94	76-79
	0.500-0.525	2	90-97	92-107	78-102
	1.02-1.05	2	92-93	101	79-81
			Ave = 98	Ave = 92	Ave = 85

[a] Parentheses = number of samples outside acceptable range.

Table 3. Recovery of triclopyr and metabolite residues from fortified fish samples using GRM 97.02 (MRID 44456103)

Matrix	Fortification (ppm)	Number of Samples	Recovery (%)		
			Triclopyr	TCP	TMP
Bluegill fillet	0.01	8	73-97	73-103	64-121 (2)
	0.03	1	72	90	67
	0.05	5	63-89	65-93	57-89 (3)
	0.13	1	70	91	65
	0.25	3	63-95	67-96	62-83 (2)
	0.50	1	80	90	76
	1.00	4	73-85	78-98	60-83 (1)
	1.50	1	67	73	83
	2.50	1	79	82	98
	3.00	1	78	82	62
	5.00	2	85-89	90-98	83-88
Catfish fillet	0.01	14	65-114	73-170	73-119
	0.05	6	63-94	65-97	69-120 (2)
	0.10	6	77-97	80-104	74-101
	0.50	4	66-81	72-89	71-85
	1.00	11	59-101	53-104	70-100
	5.00	1	91	95	99

Table 4. Concurrent method recovery of residues (MRID 44456104) from fortified water and fish samples using GC/MSD methods GRM 95.18 and GRM 97.02, respectively (TX, 1996)

Matrix	Fortification level	Number of Samples	Recovery (%)		
			triclopyr	TCP	TMP
Water	0.10 ppb	18	71-114	67-116 (1)	70-110
	0.25 ppb	3	88-119	72-114	93-118
	0.5 ppb	3	79-105	82-91	101-103
	1.0 ppb	6	73-118	81-110	82-104
	5.0 ppb	6	79-111	77-113	75-108
	10 ppb	5	76-104	78-93	80-105
	25 ppb	3	76-93	79-84	83-95
	50 ppb	3	83-106	83-103	83-105
	100 ppb	2	84-91	86-87	72-88
	250 ppb	2	93-94	90-95	93-99
	500 ppb	8	88-110	85-109	86-107
	1000 ppb	1	92	96	94
	2000 ppb	1	93	86	99
	3000 ppb	1	116	105	100
Bluegill fillet	0.01 ppm	3-4	80-128	67-113	55-73
	0.50 ppm	2	69-70	74	65-68
	2.50 ppm	2	84-113	109-138	53-68
	5.00 ppm	1-2	72-93	95-100	71
Catfish fillet	0.01 ppm	6	65-89	69-97	83-106
	5.00 ppm	3	52-98	66-102	81-87

An independent method validation (ILV) trial was reported in the following volume:

> 44456112 Emery, M. Independent Laboratory Validation of DowElanco Method GRM 97.02 - Determination of Residues of Triclopyr, 3,5,6-Trichloro-2-pyridinol, and 2-Methoxy-3,5,6-trichloropyridine in Fish tissues by Capillary Gas Chromatography with Mass Selective Detection. Protocol Number 06-9708. 109 p.

Independent validation was conducted by Minnesota Valley Testing Laboratories, Inc. (MVTL), New Ulm, MN. Following the first trial, the laboratory reported low recovery of triclopyr or

metabolites from SPE during the first trial and suggested that the method be modified to include addition of decane to the ether extract prior to SPE. It was also noted that careful attention to water bath temperature and nitrogen flow are important to the successful recovery of TMP. A third trial was needed to adequately recover TMP from crayfish inedible tissue. The results of the ILV trial are summarized in Table 5. One set of 13 samples required 33 person-hours or 3 calendar days to complete.

Table 5. Results of ILV of method GRM 97.02 for triclopyr and metabolites in fish.

Matrix	Method Trial	Fortification level (ppm)	%Recovery					
			Triclopyr		TCP		TMP	
			Values	Average ± SD	Values	Average ± SD	Values	Average ± SD
Bluegill, edible	#1	0.010	119, 104, 100, 114, 121	114±8	Compound not analyzed at this fortification (N/A)		N/A	
		0.102	98, 62, 102, 101, 79	90±20				
	#2	0.010	104, 108, 116, 113, 115	113±4	88, 90, 88, 89, 88	90±1	73, 81, 81, 65, 72	75±9
		0.102	99, 101, 105, 104, 106	105±3	88, 90, 91, 90, 88	91±1	63, 70, 61, 70, 62	66±7
	#3	0.010	N/A		N/A		73, 81, 79, 74, 78	78±4
		0.101					66, 73, 69, 72, 69	71±4
Crayfish, inedible	#1	0.010	N/A		115, 81, 107, 108, 97	103±13	N/A	
		0.102			104, 108, 107, 111, 104	109±3		
	#2	0.010	64, 74, 86, 92, 98	84±17	106, 114, 109, 112, 108	112±3	N/A	
		0.102	94, 99, 103, 106, 114	105±7	105, 102, 105, 105, 97	105±4		
	#3	0.010	N/A		N/A		70, 71, 91, 78, 78	78±11
		0.102					70, 84, 88, 89, 80	83±9

Analysis of residues in water. In the submitted aquatic studies, residues of triclopyr, TCP, and TMP in water were analyzed using GC/MS method GRM 95.18. Using this method, NaCl in 2 N HCl is added to the water sample and residues are partitioned into 1-chlorobutane. The extract is concentrated and residues are derivatized with *N*-methyl-*N*-(*tert*-butyldimethylsilyl)-

trifluoroacetamide (MBTSTFA) to form TBDMS derivatives of triclopyr and TCP. Samples are analyzed by capillary GC/MS. The LOQ is 0.1 ppb. The recovery data obtained concurrently with the field trial samples are presented in Tables 4 (above) and 6.

Triclopyr and TCP residues in water were also analyzed using an immunoassay method GRM 95.11. The LOQs were 0.145 ppb for triclopyr and 0.516 ppb for TCP. Analyses of water samples from the tests reported in MRID 44456103 using the immunoassay and GC/MSD method yielded comparable results. Recovery data for the immunoassay are presented in Table 7.

Table 6. Recovery of triclopyr, TCP, and TMP (MRID 44456102) from fortified samples of **lake water** (CA, MO, TX) using Method GRM 95.18.

Fortification (ppb)	Number of Samples	Recovery (%) [a]		
		Triclopyr	TCP	TMP
0.10	29-61	68-135 (4)	63-114 (1)	NA
0.21-0.52	16-31	96-119	76-119	68-73 n=2
1.03	20-30	90-125 (3)	81-122 (1)	NA
2.58-5.16	20	89-119	88-104	97-98 n=2
10.3	11-19	86-113	84-99	87 n=1
15.5	3	NA	83-93	NA
25.8-31.0	11-20	78-102	79-95	NA
51.6-62	4	74-100	76-90	NA
100-103	2-13	80-115	77-88	NA
199-258	15	62-92 (1)	NA	NA
498-516	7	74-85	NA	NA
748	5	75-85	NA	NA
997	6	72-88	NA	NA
1245	3	85-97	NA	NA
1496	9	67-94 (1)	NA	NA
1993	6	74-94	NA	79
2367-2491	7	81-96	NA	NA
2990-3944	11	78-96	NA	72
6311	3	78-92	NA	NA
7888	2	75-92	NA	NA
9466-10254	3	73-95	NA	NA
12621-15777	5	73-94	NA	NA
		Ave = 93	Ave = 90	Ave = 82

[a] Parentheses = number of samples outside acceptable range

Table 7. Recovery of residues in **water** (MRID 44456103) from fortified samples using the **immunoassay** GRM 95.11 for triclopyr and TCP and the GC/MSD method for TMP.

Fortification (ppb)	Number of Samples	Recovery (%)		
		Triclopyr	TCP	TMP
0.05	2	NA [a]	NA	101-104
0.10	36	56-125 (3) [b]	NA	79- 129 (1), n=50 [c]
0.40	2	NA	NA	81-82
0.50	48	NA	91-124 (4)	NA
1.00	10	87-105	105-123 (1)	87, n=1
2.00	26	NA	NA	81-101
3.00	40	NA	102-124 (3)	NA
10.00	18	NA	NA	82-101
20.00	5	74-111	NA	NA
25.00	15	NA	98-121 (1)	NA
40.00	7	NA	NA	82-101
100.00	2	101-118	102-114, n=9	NA
200.00	4	99-112	NA	NA
1000.00	18	96-125 (2)	NA	NA
2000.00	23	92-124 (1)	NA	NA
3000.00	10	88-106	NA	NA
5000.00	2	109-112	NA	NA

[a] NA = not applicable; analyte not tested at this fortification.
[b] Number of samples outside the acceptable range of 70-120% in parentheses.
[c] Number of samples if different from number in column 2.

Conclusions. GC/MS methods GRM 95.18 for triclopyr, TCP, and TMP in water and GRM 97.02 for these residues in fish and shellfish are adequate for data collection. Immunoassay method GRM 95.11 is adequate for analysis of triclopyr and TCP in water. Method GRM 97.02 has undergone a successful independent laboratory validation. A Petition Method Validation (PMV) request has been sent to ACB (D264679, W. Donovan, 07-APR-2000). The petitioner should submit the results of a radiovalidation study to ensure the ability of the analytical method to recover aged residues.

OPPTS GLN 860.1360: Multiresidue Method

The FDA PESTDATA database dated 1/94 (PAM Vol. I, Appendix I) indicates that triclopyr is completely recovered (<80%) using the multiresidue method given in PAM Vol. 1 Section 402.

Data pertaining to multiresidue methods testing of triclopyr and its metabolites through Protocols B, C, D, and E have been submitted and forwarded to FDA (Triclopyr RED, 27-OCT-1998).

OPPTS GLN 860.1380: Storage Stability Data

DowElanco submitted storage stability data to support the fish and shellfish field trials:

> 45170901 Foster, D.R. (2000) Frozen Storage Stability of Triclopyr, Pyridinol, and Methoxypyridine in Fish and Crayfish Tissues. Laboratory Study ID RES96092. Unpublished study submitted by Dow AgroSciences. 207 p.

Untreated samples of bluegill, catfish and crayfish fillets were fortified with triclopyr, TCP, and TMP each at 0.1 ppm. Samples were placed in frozen storage at -20 C and analyzed on day zero and at intervals up to 570 days (19 months) for crayfish and up to 2 years for fish. Residues were analyzed using Method GRM 97.02, described above, with a LOD of 0.003 ppm and a LOQ of 0.01 ppm for each analyte. The method is adequate for data collection.

The results of residue analyses after storage, along with fresh sample fortification recoveries and stored sample recoveries corrected for fresh recoveries, are presented in Table 8. Residues of triclopyr and its metabolites TCP and TMP were stable in crayfish edible portions for up to 19 months and in bluegill fillet for up to 2 years. Residues of each compound in catfish were analyzed after up to 168 days and were stable for that period.

These data support the fish and shellfish residue trials described below under OPPTS GLN 860.1400, in which samples were stored for up to 21 months.

Table 8. Storage stability of triclopyr and metabolites in fish and crayfish edible fillets, fortified with each analyte at 0.1 ppm, in frozen storage (-20 C).

Matrix	Storage (days)	Triclopyr			TCP			TMP		
		% rec. stored	% fresh rec	% corr stored	% rec. stored	% fresh rec	% corr stored	% rec. stored	% fresh rec	% corr stored
Crayfish	0	87, 88	96	92	80, 84	80	103	64, 63	80	80
	92	97, 97	84	115	90, 85	77	114	81, 85	80	103
	212	103, 95	100	99	106, 107	92	116	82, 94	88	100
	570	99, 109	96	108	90, 102	90	107	87, 79	86	97
Bluegill	0	87, 85	93	94	87, 85	98	88	50, 64	64	89
	64	88, 100	87	109	91, 89	99	91	74, 69	72	99
	148	99, 91	84	113	94, 88	95	96	78, 72	82	91
	400	91, 98	79	120	98, 78	83	106	88, 84	89	97

Matrix	Storage (days)	Triclopyr			TCP			TMP		
		% rec. stored	% fresh rec	% corr stored	% rec. stored	% fresh rec	% corr stored	% rec. stored	% fresh rec	% corr stored
	780	120, 119	104	115	92, 92	96	96	70, 80	73	102
Catfish	0	93, 56	80	94	100, 92	98	98	80, 81	88	92
	71	92, 103	83	118	119, 100	95	111	66, 70	83	82
	161	63, 94	72	111	60, 54	66	86	95, (48)	99	96
	168	NA	NA	NA	NA	NA	NA	79, 87	82	99

OPPTS GLN 860.1400: Water, Fish, and Irrigated Crops

Metabolism in Fish

Dow Chemical Co. submitted a metabolism study of pyridine-labeled [^{14}C]triclopyr in fish. The study was conducted at Dow Chemical Co. in Midland, MI, and is reported in the following volume:

44015101 Rick, D.L., H.D. Kirk, D.D. Fontaine, M.J. Bartels, K.B. Woodburn (1996) The Nature of Triclopyr Residues in the Bluegill, *Lepomis macrochirus* Rafinesque. Study ID DECO-ES-2761. Unpublished study submitted by Dow Chemical Co. 64 p.

[2,6-pyridine-^{14}C]Triclopyr, 24.12 mCi/mmole, radiochemical purity >99%, diluted with unlabeled triclopyr to a final specific activity of 13.4 dpm/ng, was added to Lake Huron feed water in 43-L aquaria. Water quality parameters measured were: pH 7.4-7.7, alkalinity 43-70 mg/L as $CaCO_3$, hardness 40-74 mg/L as $CaCO_3$, conductivity 210-260 μmhos/cm. The target [^{14}C]triclopyr concentration was 7.5 mg/L (actual, 7.1-7.7 mg/L), 3x the registered aquatic use rate. Two aquaria were prepared, one treated and the other as a control. Juvenile bluegill, *Lepomis macrochirus*, were acclimated for 31 days and 125 fish (mean weight 0.712 g at the beginning of treatment) were placed in each aquarium. Fish were sampled on days 1, 2, 4, 7, and 10 for radioactivity in whole fish; on days 7 and 10 samples were also taken for dissection and for extraction and HPLC characterization of ^{14}C-residues.

Total radioactive residues.

Whole fish, fillet muscle tissue, inedible remainder tissues, and solids from extraction were tissue-solubilized and the radioactive residues were analyzed by LSC. Radioassay limits of detection were 0.003 ppm for fish and tissues and 0.001 ppm for HPLC fractions. Total radioactivity in whole fish, fillet, and remainder tissue is presented in Table 9.

Total radioactive residues in whole fish remained constant at ~5.0-6.3 ppm throughout the exposure period. After 14 days of depuration, radioactivity had decreased in whole fish by 88%. ^{14}C-Residues in the edible fillet muscle was ~10% of the whole-fish concentration at 7 days and 6% at 10 days.

The petitioner stated that the bioconcentration factors were 0.77 mL/g in whole fish, 0.06 and 0.83 mL/g in muscle and inedible portions, respectively. The elimination half-life was calculated at 0.6 days.

Extraction and characterization of ^{14}C-residues

Whole fish were frozen for up to 5 days prior to extraction and analyzed by HPLC 2-3 days later. Duplicate frozen fish from 7 and 10 days exposure were thawed and the residues were extracted with acetonitrile (ACN) containing 1% phosphoric acid. The extract volume was reduced by rotary evaporation, the residues were reconstituted in methanol:water:ACN (5:5:2), and the extract was acidified to pH 2-3 with 85% phosphoric acid. Of the total radioactivity in fish, 92-95% was extracted.

Residues were characterized using a reverse-phase HPLC in a system comprised of a C_{18} column and a mobile gradient of water/ACN. Peaks were detected by a UV monitor and radioactivity was measured using a flow-through detector. The results of metabolite characterization are presented in Table 10.

Triclopyr and TCP were identified by comparison of retention times with standards and the identities were confirmed by LC/MS. A TCP conjugate was identified following hydrolysis of an aliquot of fish extract with 1 N NaOH at 80 C for 2 hours; subsequent HPLC analysis showed disappearance of the conjugate peak and corresponding increase in free TCP. The taurine conjugate of triclopyr was characterized by LC/MS and the identity verified by co-chromatography with a synthesized taurine-triclopyr standard. Triclopyr accounted for 24-26% TRR in whole fish exposed for 7 and 10 days. TCP, the TCP-conjugate, and the taurine-triclopyr conjugate accounted for 24, 19, and 16% TRR in the 7-day sample and 9, 34, and 16% in the 10-day sample.

A non-polar component, peak #5 accounted for ~9% TRR. The potential metabolite TMP was sought by subjecting the HPLC extract to partitioning with cyclohexane; subsequent HPLC detected TMP at 0.025 ppm (0.5% TRR), <10% of the peak #5 residue concentration. Also ruled out were the methyl ester of triclopyr and 3,5-dichloro-6-methylthio-2-pyridinol. The petitioner reported remaining HPLC peaks comprising a total of 6.6-8.5% TRR, although a complete quantitation of all HPLC peaks was not reported. Identified components, peak #5, the total of components designated as "other," and calculated non-extracted fractions accounted for 81-83% TRR; an accounting of the remaining 18-19% was not given.

Conclusions

Bluegill sunfish were exposed to pyridine-labeled [^{14}C]triclopyr at a nominal concentration of 7.5 ppm (3x the proposed rate) for 10 days. The total radioactive residues (TRR) were 0.515 and 0.369 ppm in edible fillet muscle after 7 and 10 days of exposure, respectively [10 and 6% of the radioactivity in whole fish (5.018 and 6.029 ppm)]. Residues did not concentrate in fish relative to the concentration in water of 7.1-7.7 ppm. From HPLC analysis of ^{14}C-residues in whole fish, triclopyr accounted for 24-26% TRR in whole fish exposed for 7 and 10 days. TCP, the TCP-conjugate, and the taurine-triclopyr conjugate accounted for 24, 19, and 16% TRR in the 7-day sample and 9, 34, and 16% in the 10-day sample. A peak designated #5 was analyzed for TMP and two other possible non-polar components, but these were not found. Of the TRR, 74-76% were characterized. According to this study, the major residues of triclopyr in fish are the parent compound and TCP and their conjugates.

HED notes that the metabolic profile of triclopyr in fish from this metabolism study is not consistent with the results reported in the magnitude of the residue (MOR) studies. Specifically, this study found TMP to comprise <1% TRR, while in the MOR studies TMP was consistently found to be the primary residue in bluegill fillet samples after exposure to triclopyr-treated water for 1 day or more. The MOR studies analyzed edible fish tissues using an analytical method that successfully passed ILV and was demonstrated to successfully recover TMP residues from fish samples (80-101% recovery over a fortification range of 0.010 - 5.00 ppm). In contrast, the fish metabolism study analyzed whole fish samples using a different extraction procedure that was not subjected to ILV and gave a mean extraction efficiency of TMP-fortified fish samples of just 60 ± 10% over an unspecified range. Thus, HED finds that the weight-of-the-evidence favors the results of the MOR studies that suggest the residues of concern in triclopyr-treated fish are the parent, TCP, and TMP.

The HED MARC determined that the residues of concern in fish and shellfish are triclopyr, TCP, and TMP (D274243, W. Donovan and W. Dykstra, 27-APR-2001). The Committee also determined that the residues of concern in drinking water are triclopyr and TCP.

Table 9. Total radioactive residues in whole fish, fillet muscle, and inedible remains of bluegill sunfish exposed to [^{14}C]triclopyr for up to 10 days at 7.1-7.5 ppm.

Study day	Total radioactive residues (ppm)		
	Whole fish	Fillet muscle	Remains
Exposure period			
1	6.340	Not analyzed	Not analyzed
2	6.251		
4	5.027		
7	5.018	0.515	6.060
10	6.029	0.369	6.175

Study day	Total radioactive residues (ppm)		
	Whole fish	Fillet muscle	Remains
Clearance period			
1	4.264	Not analyzed	Not analyzed
4	1.047		
7	0.864		
11	0.784		
14	0.755		

Table 10. Characterization of ^{14}C-residues in whole fish exposed to [^{14}C]triclopyr for 7 and 10 days at 7.1-7.5 ppm.

	7-Day (TRR = 5.018 ppm)		10-Day (TRR = 6.029 ppm)	
Metabolite/fraction	% TRR	ppm	% TRR	ppm
Triclopyr	24.2	1.215	26.0	1.568
TCP	23.6	1.184	9.0	0.543
TCP-conj	18.8	0.944	34.0	2.047
Taur-conj	15.7	0.788	15.7	0.947
Peak 5	9.2	0.462	8.8	0.531
Other	8.5	0.427	6.6	0.395
Total accounted	100.0	5.020	100.1	6.031

Magnitude of the Residue Studies

The petitioner provided three submissions encompassing five studies of the aquatic dissipation of triclopyr and its metabolite TCP and TMP and residues of these compounds in fish and shellfish. A protocol for these studies was approved by HED (D210628, G. Otakie, 20-APR-1995). The results of these studies are presented in the following volumes:

44456102 Houtman, B.A. (1997) Aquatic Dissipation of Triclopyr in Lake Minnetonka, Minnesota. Laboratory Study ID: ENV94001. Unpublished study submitted by DowElanco. 527 p.

44456103 Foster, D.A., K.D. Getsinger, and D.G. Petty (1997) The Aquatic Dissipation of Triclopyr in a Whole-Pond Treatment. Laboratory Study ID: ENV95012. Unpublished study submitted by DowElanco. 306 p.

44456104 Foster, D.A., K.D. Getsinger, and D.G. Petty (1997) Triclopyr Dissipation and the formation and decline of its TMP and TCP Metabolites in a Aquatic Environment. Laboratory Study ID: ENV96052. Unpublished study submitted by DowElanco. 259 p.

MRID 44456102. Minnesota lake study, 1994. Triclopyr aquatic testing was conducted in Lake Minnetonka, MN, a complex of 15 morphologically distinct basins with a total volume of 400.6 million m^3. Three rectangular plots were established in separate bays of the lake. Phelps Bay and Carsons Bay plots were treated. Each plot was divided into quadrants with sampling stations in the center of each quadrant and at the center of the plot. Additional off-plot sampling stations were set up at 100, 400, 800, and 1600 m from the edge of the plot. Test fish and shellfish species consisted of 224 largemouth bass (*Micropterus salmoides*), 301 bluegill (*Lepomis macrochirus*); 36 bullhead catfish (*Ictalurus nebulosus.*); 385 white suckers (*Catostomus commersoni*); 1075 crayfish (*Orocnectes virlis* or *O. immunis*); and 400 mussels (*Lampsillis silliquaoidea*) also reported as clams. In one plot, designated as Phelps Bay, triclopyr 3 lb ae/gal was applied below the water surface using a boat-mounted stray boom with three trailing hoses 1.2, 2.4, and 3.7 m long, to a target concentration of 2.5 ppm. The second plot, in Carsons Bay, was surface-treated using a boat-mounted Radiarc (boomless, low volume) sprayer. The fluorescent dye rhodamine WT was tank-mixed and delivered with the test substance. The petitioner indicated that excellent correlation was observed between the dispersion and dissipation of the dye with that of triclopyr.

Water and fish were sampled at 1 hour after treatment, at 3, 6, and 12 hours, at 1, 2, 3, and 5 days, and 1, 2, 3, 4 weeks; water was also sampled at 6 weeks. Water was refrigerated and analyzed 7 months after sampling using GC/MSD method GRM 95.18; the calculated LOD was <0.10 ppb and the calculated LOQs were 0.145 ppb for triclopyr and 0.107 ppb for TCP. Analyses were conducted at DowElanco, Indianapolis, IN.

Fish and shellfish samples were refrigerated and separated into edible and inedible portions and shipped frozen to the analytical laboratory, Dow Chemical H&ES, Midland, MI, and DowElanco, Indianapolis, IN. Residues were analyzed using the GC/MS method, GRM 97.02, described above. Calculated LODs were 0.005 and 0.006 ppm for fish and shellfish, respectively, and LOQs were ≤0.017 and ≤0.018 ppm. Residue analyses were completed within 14 months of sample collection.

Triclopyr and metabolite residues in lake water, fish and shellfish are presented in Tables 11 through 17. The maximum total residue of triclopyr, TCP, and TMP in water from the treated plots (Table 11) was 3.33-3.73 ppm, 3.32-3.70 ppm of which was parent, reached at 3 hours after treatment. After 5 days, combined residues were 0.7 and 1.8 ppm; residues fell below 0.5 ppm after 1 week in Phelps Bay and 2 weeks in Carsons Bay. After 2 weeks, Residues declined by >98% to 0.056 ppm in Phelps Bay after 2 weeks and to 0.064 ppm after 4 weeks in Carsons Bay. The maximum off-plot combined residue level was 0.294 ppm 100 m from the Carsons Bay plot. Throughout the study, TCP and TMP residue levels were very low compared to parent.

Maximum combined residues in edible bass tissue (Table 12) were 0.952 and 0.670 ppm, respectively, in Phelps bay and Carsons Bay, after 3 days. TMP accounted for >94% of the combined residue at 3 days, with only <1 to 4% parent or TCP. In Carsons Bay, combined residues were <0.06 ppm at the end of the 4-week study.

Maximum combined residues in edible bluegill tissue (Table 13) were 0.455 and 0.754 ppm, respectively, in Phelps bay and Carsons Bay, after 3 days. At 3 days 95% of the combined residue consisted of TMP, and only minor amounts of parent and TCP were present. At the end of the 4-week study, combined residues were <0.015 and <0.047 ppm.

Maximum combined residues in edible bullhead tissue (Table 14) were 0.275 ppm in Carsons Bay, after 1 week. TMP was the most abundant residue (81% of combined at 1 week). Combined residues were <0.04 ppm at the end of the 4-week study.

Maximum combined residues in edible sucker tissue (Table 15) were 0.463 and 0.678 ppm, respectively, in Phelps bay and Carsons Bay, after 1 or 3 days. Most of the combined residue consisted of TMP, and only minor amounts of parent and TCP. Combined residues were <0.061 ppm at the end of the 4-week study.

Maximum combined residues in edible clam tissue (Table 16) were 0.405 and 0.523 ppm, respectively, in Phelps Bay and Carsons Bay, after 3 days. At early time points, triclopyr was the predominant compound, then after 3 days most of the combined residue consisted of TMP. Combined residues were 0.07 ppm at the end of the 4-week study in Carsons Bay, and were <0.018 ppm in Phelps Bay.

Maximum combined residues in edible crayfish tissue (Table 17) were 0.310 and 0.454 ppm, respectively, in Phelps bay and Carsons Bay, after 3 days. At early time points, triclopyr was the predominant compound. Combined residues were 0.044 ppm at the end of the 4-week study in Carsons Bay, and were <0.030 ppm in Phelps Bay.

Table 11. Residues of Triclopyr, TCP, and TMP in **water** from two locations in Lake Minnetonka, MN (MRID 44456102).

Sampling period	Residue concentration (ppm), Mean [a]			
	Triclopyr	TCP	TMP	Total
Phelps Bay				
1 hour	1.937	0.019	<0.0001	1.956
3 hour	3.701	0.024	<0.0001	**3.725**
6 hour	2.631	0.021	0.001	2.653
12 hour	2.254	0.013	0.002	2.269
1 day	2.844	0.015	0.004	2.863
2 day	2.090	0.008	0.003	2.101
3 day	1.913	0.003	0.004	1.920
5 day	0.718	0.002	<0.0001	0.720
1 week	0.459	0.002	<0.0001	0.461
2 week	0.056	0.0002	<0.0001	0.056
3 week	0.019	<0.0001	<0.0001	0.019
4 week	0.008	<0.0001	<0.0001	0.008
6 week	0.002	<0.00003	<0.0001	0.002
Carsons Bay				
1 hour	3.072	0.020	<0.0001	3.092
3 hour	3.318	0.014	<0.0001	**3.332**
6 hour	2.861	0.013	0.001	2.895
12 hour	2.530	0.011	0.002	2.543
1 day	2.265	0.007	0.002	2.274
2 day	2.370	0.008	0.004	2.382
3 day	2.118	0.008	0.004	2.130
5 day	1.809	0.007	<0.0001	1.816
1 week	1.419	0.004	<0.0001	1.423
2 week	0.386	0.005	<0.0001	0.391
3 week	0.163	0.002	<0.0001	0.165
4 week	0.062	0.002	<0.0001	0.064
6 week	0.009	0.0002	<0.0001	0.009

[a] Each value is the mean of duplicate samples taken at three depths at each of 5 sampling stations in the treated plot.

Table 12. Residues in of triclopyr and metabolites in **bass, edible tissue** from Lake Minnetonka, MN plots treated to a concentration of 2.5 ppm (44456102).

Sampling period	Residues (ppm); mean of duplicate samples			
	Triclopyr	TCP	TMP	Total
Phelps Bay				
1 hour	<0.005	<0.005	<0.005	<0.015
3 hour	<0.017	<0.005	0.010	<0.032
6 hour	<0.005	<0.005	0.129	<0.139
12 hour	<0.005	<0.005	0.537	<0.547
1 day	<0.005	<0.017	0.719	<0.741
3 day	<0.005	0.018	0.929	**<0.952**
1 week	<0.005	0.024	0.519	<0.548
Carsons Bay				
1 hour	0.037	<0.005	<0.005	<0.047
3 hour	<0.017	<0.005	<0.017	<0.039
6 hour	<0.017	<0.005	0.025	<0.047
12 hour	<0.005	<0.005	0.059	<0.069
1 day	<0.017	<0.017	0.155	<0.189
3 day	0.024	0.017	0.629	**0.670**
1 week	<0.005	0.010	0.208	<0.223
2 week	<0.005	<0.017	0.103	<0.125
3 week	<0.005	<0.005	0.069	<0.079
4 week	<0.005	0.001	0.051	<0.057

Table 13. Residues in of triclopyr and metabolites in **bluegill, edible tissue** from Lake Minnetonka, MN plots treated to a concentration of 2.5 ppm (44456102).

Sampling period	Residues (ppm); mean of duplicate samples			
	Triclopyr	TCP	TMP	Total
Phelps Bay				
1 hour	<0.017	<0.005	<0.005	<0.027
3 hour	<0.017	<0.005	0.012	<0.036
6 hour	<0.017	<0.005	0.123	<0.145
12 hour	<0.017	<0.017	0.157	<0.191
1 day	0.026	<0.017	0.210	<0.254
3 day	<0.005	0.017	0.433	**<0.455**
1 week	<0.005	<0.017	0.111	<0.133
2 week	<0.005	<0.017	0.038	<0.061
3 week	<0.005	<0.005	<0.017	<0.027
4 week	<0.005	<0.005	<0.005	<0.015
Carsons Bay				
1 hour	<0.017	<0.005	<0.017	<0.040
3 hour	<0.017	<0.005	0.025	<0.047
6 hour	0.017	<0.005	0.020	<0.042
12 hour	<0.017	<0.017	0.104	<0.138
1 day	<0.017	<0.017	0.226	<0.260
3 day	<0.017	0.023	0.714	**<0.754**
1 week	<0.017	<0.017	0.296	<0.330
2 week	<0.005	<0.017	0.244	<0.267
3 week	<0.005	<0.005	0.029	<0.039
4 week	<0.005	<0.017	0.025	<0.047

Table 14. Residues in of triclopyr and metabolites in **bullhead, edible tissue** from Lake Minnetonka, MN plots treated to a concentration of 2.5 ppm (44456102).

Sampling period	Residues (ppm); mean of duplicate samples			
	Triclopyr	TCP	TMP	Total
Carsons Bay				
1 hour	0.008	<0.017	<0.005	<0.030
3 hour	0.013	<0.005	<0.005	<0.023
6 hour	0.023	<0.0017	<0.017	<0.047
12 hour	0.013	0.018	0.049	0.080
1 day	0.022	0.014	0.059	0.095
3 day	0.013	0.032	0.173	0.218
1 week	0.010	0.043	0.222	**0.275**
2 week	<0.005	0.013	0.086	<0.104
3 week	<0.005	0.006	0.049	<0.060
4 week	<0.005	<0.017	0.016	<0.038

Table 15. Residues in of triclopyr and metabolites in **sucker, edible tissue** from Lake Minnetonka, MN plots treated to a concentration of 2.5 ppm (44456102).

Sampling period	Residues (ppm); mean of duplicate samples			
	Triclopyr	TCP	TMP	Total
Phelps Bay				
1 hour	0.012	0.012	<0.005	<0.029
3 hour	0.019	<0.017	<0.017	<0.053
6 hour	0.024	<0.017	0.020	<0.061
12 hour	0.036	0.008	0.148	0.192
1 day	0.051	0.036	0.376	**0.463**
3 day	0.040	0.025	0.232	0.297
1 week	0.020	0.020	0.083	0.123
2 week	<0.005	<0.017	0.029	0.041
3 week	<0.005	<0.017	<0.017	<0.039
4 week	<0.005	<0.017	<0.017	<0.039
Carsons Bay				
1 hour	0.021	<0.017	<0.005	<0.043
3 hour	0.041	<0.017	<0.017	<0.075
6 hour	0.041	0.007	0.058	0.106
12 hour	0.053	0.020	0.265	0.338
1 day	0.061	0.021	0.405	0.487
3 day	0.043	0.021	0.614	**0.678**
1 week	0.044	0.016	0.175	0.235
2 week	0.012	<0.017	0.125	<0.154
3 week	<0.017	<0.017	0.044	<0.078
4 week	<0.005	0.010	0.046	<0.061

Table 16. Residues in of triclopyr and metabolites in **clam, edible tissue** from Lake Minnetonka, MN plots treated to a concentration of 2.5 ppm (44456102).

Sampling period	Residues (ppm); mean of duplicate samples			
	Triclopyr	TCP	TMP	Total
Phelps Bay				
1 hour	<0.018	<0.006	<0.006	<0.030
3 hour	0.095	<0.018	<0.018	<0.131
6 hour	0.126	0.006	<0.018	<0.150
12 hour	0.120	0.013	0.014	0.147
1 day	0.146	0.012	0.052	0.210
3 day	0.131	0.012	0.262	**0.405**
1 week	0.040	<0.018	0.050	<0.096
2 week	<0.018	<0.006	0.017	<0.041
3 week	<0.018	<0.006	<0.018	<0.042
4 week	<0.006	<0.006	<0.006	<0.018
Carsons Bay				
1 hour	0.074	0.009	<0.006	<0.089
3 hour	0.134	0.019	<0.006	<0.159
6 hour	0.158	0.013	0.018	0.179
12 hour	0.154	0.011	0.036	0.201
1 day	0.153	0.013	0.132	0.298
3 day	0.164	0.015	0.344	**0.523**
1 week	0.156	0.020	0.268	0.444
2 week	0.055	0.008	0.113	0.176
3 week	0.063	0.008	0.052	0.123
4 week	0.032	0.020	0.018	0.070

Table 17. Residues in of triclopyr and metabolites in **crayfish, edible tissue** from Lake Minnetonka, MN plots treated to a concentration of 2.5 ppm (44456102).

Sampling period	Residues (ppm); mean of duplicate samples			
	Triclopyr	TCP	TMP	Total
Phelps Bay				
1 hour	<0.018	<0.006	<0.006	<0.030
3 hour	0.042	<0.018	<0.006	<0.066
6 hour	0.041	<0.018	<0.018	<0.077
12 hour	0.090	0.007	<0.018	<0.115
1 day	0.179	0.011	0.042	0.232
3 day	0.163	0.028	0.119	**0.310**
1 week	0.085	0.030	0.057	0.172
2 week	0.020	<0.018	<0.018	<0.056
3 week	0.016	<0.018	<0.006	<0.040
4 week	<0.018	<0.006	<0.006	<0.030
Carsons Bay				
1 hour	0.068	<0.018	<0.006	<0.092
3 hour	0.032	<0.006	<0.006	<0.044
6 hour	0.073	<0.018	0.012	<0.103
12 hour	0.053	0.007	0.049	0.109
1 day	0.108	0.014	0.083	0.205
3 day	0.179	0.031	0.244	**0.454**
1 week	0.157	0.037	0.206	0.400
2 week	0.052	0.013	0.049	0.114
3 week	0.058	0.013	0.033	0.104
4 week	<0.018	0.008	<0.018	<0.044

MRID 44456103 - Pond studies in CA, MO, and TX. Three pond trials were conducted in CA, MO, and TX. Two treated ponds and one control pond were sampled at each location. In each treated pond were three sampling stations. At each time point, upper and lower portions of the pond were sampled once and sometimes twice, at each station, resulting in six to eight samples per interval at each location. The two test fish species were bluegill (*Lepomis macrochirus*) and catfish (*Ictalurus sp.*).

The CA site was located at the CA Department of Fish and Game Aquatic Toxicology Lab and facilities near Elk Grove in Sacramento County. Two treated 0.30 acre ponds (A and B) each had a total volume of 984.5 m^3. Triclopyr 3 lb ae/gal triethanolamine formulation was applied at 6.8 L in 23 L of water to reach a target concentration of 2.5 ppm, using a boat-mounted power sprayer with a hand wand releasing the test material at or slightly below the water surface. A total of 170 bluegill and 125 catfish were placed in cages in the treated ponds and 60 of each species were placed in the control pond. Water and fish were sampled one, 6, and 12 hours after treatment and on days 2, 5, and 7.

The MO test was conducted at the U.S. Geological Survey Environmental Contaminants Research Center near Columbia, MO. The two treated ponds averaged 792 m^3 in volume. The test material was applied by hand spraying from a truck driven around the circumference of each pond. A concentration of 2.5 ppm was achieved using 4.3-6.2 L of the 3 lb/gal ae product in 95 L of water. A total of 250 bluegill and 130 catfish were placed in cages in the treated ponds. The control pond contained 322 bluegill and 180 catfish. Water and fish were sampled one, 3, and 12 hours after treatment and on days 1, 5, 7, and 14.

In TX the test site was the USAE Waterways Experimental Station, Lewisville Aquatic Ecosystem Research Facility, near Lewisville, TX. The ponds were treated by spraying 19 L of test substance diluted in 95 L of water onto the pond surface from the circumference to reach a concentration of 2.5 ppm. A total of 150 bluegill and 170 catfish were placed in cages in two test ponds averaging 2880 m^3; 80 bluegill and 150 catfish were placed in a control pond. Water and fish were sampled one, 3, and 12 hours after treatment and after 1 day, 5 days, 1 week, and 2 weeks.

Water and fish tissue samples were refrigerated in the field. Fish were processed in a facility near each test location and shipped frozen to DowElanco, Indianapolis, IN. Fish tissues and some water samples were analyzed by Quality Management and Analytical Services (QMAS), Walhalla, ND, and water analyses were also conducted at DowElanco Global Environmental Chemistry Laboratories in Indianapolis, IN. Analyses of water and fish samples was completed approximately 1 year after sampling. Triclopyr and TCP in water were analyzed using immunoassay method GRM 95.11. Reanalysis of triclopyr and TCP in representative water samples using GC/MSD method GRM 95.18 gave results comparable to those from the immunoassay. TMP in water was analyzed using method GRM 95.18. Residues in fish tissues were analyzed using method GRM 97.02. The maximum calculated LOQ for water was 0.516 ppb of TCP (0.0005 ppm). The LOD for water was 0.010 ppb. The maximum calculated LOD and LOQ for fish were 0.007 ppm and 0.023 ppm, respectively.

The results of residue analysis in water and fish from the CA, MO, and TX sites are presented in Tables 18 through 26. In these tables, each value represents the average of six to eight samples and the data are corrected for method recovery (percentages not specified), as presented in the submission. For this review, total residues of the three analytes were calculated. For data reported in the submission as ND or NQ in fish, the LOD or LOQ value was added in the total. For water, the LOQ was added, but residues below the LOD (<0.00001 ppm) were not.

Overall, results from the three locations were similar. Total triclopyr, TCP, and TMP residues in water attained maximum levels of 2.1-2.8 ppm, at 1-3 hours posttreatment in CA (Table 18), 1 day in MO (Table 21), and 6 hours in TX (Table 24). Total residues decreased to levels below 0.5 ppm after 3 weeks in all sites with the exception of one CA pond, in which the 3-week level was 0.645 ppm with residues falling below 0.5 ppm after 4 weeks. Throughout the tests, little degradation of triclopyr in water was observed; maximum TCP levels were 0.004-0.022 ppm and maximum TMP concentration was 0.004-0.007 ppm.

Maximum total residues in bluegill fillet were <0.439-0.956 ppm, reached after 1-2 weeks in CA (Table 19) and MO (Table 22) and after 0.5-3 days in TX (Table 25). Maximum total residues in catfish fillet (Tables 20, 23, and 26) were <0.755-<2.971 ppm, reached after intervals of 12 hours to 2 weeks. Data on fish viscera (not presented here) show decreases in triclopyr over time with concurrent increases in metabolite levels, whereas, in fillet tissues, TMP levels were substantially higher than those of the other analytes. The data on both species indicate that triclopyr is metabolized by fish to TCP and subsequently to TMP, and that TMP is the residue transferred to muscle tissue, accumulating for no longer than 2 weeks.

Table 18. Summary of average triclopyr and metabolite residues in **pond water**, treated to a target concentration of 2.5 ppm, from the **California** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total
Water Pond A				
1 hour	2.087	0.012	ND [b]	**2.099**
3 hours	1.934	0.010	ND	1.944
6 hours	1.730	0.010	ND	1.740
10 hours	1.666	0.010	0.001	1.677
1 day	1.935	0.005	0.002	1.942
2 days	1.792	0.005	0.003	1.800
3 days	1.801	0.005	0.003	1.809
5 days	1.647	0.008	0.004	1.659
1 week	1.523	0.007	0.004	1.534
2 weeks	1.021	0.003	0.002	1.026
3 weeks	0.320	0.001	ND	0.321
4 weeks	0.120	ND	<0.001	<0.121
6 weeks	0.002	ND	ND	0.002
12 weeks	0.001	ND	ND	0.001
Water Pond B				
1 hour	2.520	0.017	ND	2.537
3 hours	2.663	0.017	ND	**2.680**
6 hours	2.189	0.015	ND	2.204
10 hours	1.818	0.009	0.002	1.829
1 day	2.083	0.010	0.002	2.095
2 days	2.083	0.009	0.003	2.095
3 days	2.025	0.004	0.003	2.032
5 days	1.700	0.007	0.004	1.711
1 week	1.588	0.010	0.003	1.601
2 weeks	1.307	0.004	0.003	1.314
3 weeks	0.641	0.002	0.002	0.645
4 weeks	0.193	<0.001	<0.001	<0.195
6 weeks	0.003	ND	ND	0.003
12 weeks	0.003	ND	ND	0.003

[a] Each value represents the average of 6-8 samples.
[b] ND = <0.0001 ppm; this figure was not added to totals.

Table 19. Summary of average triclopyr and metabolite residues in **bluegill fillet** from ponds treated to a target concentration of 2.5 ppm, from the **California** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Bluegill Fillet Pond A				
1 hour	<0.023	<0.007	<0.023	<0.053
3 hours	<0.023	<0.007	0.079	<0.109
6 hours	NS	NS	NS	NS
10 hours	0.032	<0.007	0.237	<0.276
1 day	0.025	<0.007	<0.023	<0.055
3 days	<0.023	0.016	0.114	<0.153
1 week	<0.023	0.016	0.326	<0.365
2 weeks	<0.007	0.016	0.546	**<0.569**
3 weeks	<0.007	<0.007	0.206	<0.220
4 weeks	<0.007	<0.007	0.060	<0.074
Bluegill Fillet Pond B				
1 hour	<0.023	<0.007	<0.023	<0.053
3 hours	0.020	<0.007	0.138	<0.165
6 hours	0.016	<0.007	0.161	<0.183
10 hours	0.037	<0.023	0.376	<0.436
1 day	0.019	<0.007	<0.023	<0.049
3 days	<0.023	0.014	0.264	<0.301
1 week	<0.023	0.015	0.789	0.827
2 weeks	<0.007	0.022	0.927	**<0.956**
3 weeks	<0.007	<0.023	0.082	<0.102
4 weeks	<0.007	<0.007	0.223	<0.237

[a] Each value represents the average of 3-4 samples.

[b] Residues <LOD or <LOQ are added into totals as <0.007 and <0.023 ppm, respectively.

Table 20. Summary of average triclopyr and metabolite residues in **catfish fillet** from ponds treated to a target concentration of 2.5 ppm, from the **California** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Catfish Fillet Pond A				
1 hour	<0.023	<0.007	0.131	<0.163
3 hours	<0.023	<0.007	0.005	<0.035
6 hours	<0.023	<0.007	0.086	<0.118
10 hours	<0.023	<0.023	0.227	<0.273
1 day	<0.007	<0.023	0.558	<0.585
3 days	<0.007	<0.023	0.725	<0.755
1 week	<0.007	<0.023	0.288	<0.318
2 weeks	<0.007	<0.007	0.361	<0.375
3 weeks	<0.007	<0.007	0.221	<0.235
4 weeks	<0.007	<0.007	0.940	**<0.954**
Catfish Fillet Pond B				
1 hour	<0.023	<0.007	0.068	<0.098
3 hours	<0.023	<0.023	0.017	<0.063
6 hours	<0.023	<0.007	0.054	<0.084
10 hours	<0.007	<0.023	0.126	<0.156
1 day	<0.007	<0.023	0.646	<0.676
3 days	<0.023	<0.023	0.758	<0.804
1 week	<0.023	<0.023	0.841	**<0.887**
2 weeks	<0.023	<0.007	0.251	<0.281
3 weeks	<0.007	<0.007	0.519	<0.533
4 weeks	<0.007	<0.007	0.269	<0.283

[a] Each value represents the average of 3-4 samples.

[b] Residues <LOD or <LOQ are added into totals as <0.007 and <0.023 ppm, respectively.

Table 21. Summary of average triclopyr and metabolite residues in **pond water**, treated to a target concentration of 2.5 ppm, from the **Missouri** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total
Water Pond A				
1 hour	1.816	0.011	NA	1.827
3 hours	1.664	0.004	ND [b]	1.668
6 hours	2.288	0.004	0.003	2.295
12 hours	2.505	0.003	0.005	2.513
1 day	2.799	0.005	0.005	**2.809**
2 days	2.467	0.004	0.005	2.476
3 days	1.909	0.004	0.006	1.919
5 days	1.712	0.003	0.007	1.722
1 week	1.407	0.007	0.006	1.420
2 weeks	0.734	0.007	0.004	0.745
3 weeks	0.228	0.001	0.001	0.230
4 weeks	0.067	<0.001	<0.001	<0.069
6 weeks	0.007	ND	ND	0.007
12 weeks	<0.001	ND	ND	<0.001
Water Pond B				
1 hour	1.170	0.004	ND	1.174
3 hours	1.977	0.003	0.001	1.981
6 hours	2.281	0.002	0.005	2.288
12 hours	2.226	0.002	0.006	2.234
1 day	2.345	0.003	0.005	**2.353**
2 days	1.749	0.003	0.006	1.758
3 days	1.486	0.002	0.007	1.495
5 days	1.386	0.002	0.006	1.394
1 week	1.045	0.004	0.007	1.056
2 weeks	0.502	0.002	0.004	0.508
3 weeks	0.137	0.001	0.001	0.139
4 weeks	0.032	ND	<0.001	<0.033
6 weeks	0.004	ND	ND	0.004
12 weeks	ND	ND	ND	ND

[a] Each value represents the average of 6-8 samples.
[b] ND = <0.0001 ppm; this figure was not added to totals.

Table 22. Summary of average triclopyr and metabolite residues in **bluegill fillet** from ponds treated to a target concentration of 2.5 ppm, from the **Missouri** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Bluegill Fillet Pond A				
1 hour	0.050	<0.007	0.006	<0.063
3 hours	0.021	<0.007	0.020	<0.048
6 hours	0.048	<0.007	0.048	<0.103
12 hours	<0.023	<0.007	0.114	<0.144
1 day	0.026	<0.023	0.134	<0.183
3 days	<0.023	0.023	0.551	<0.597
1 weeks	<0.023	0.024	0.524	<0.571
2 weeks	<0.007	<0.023	0.665	**<0.695**
3 weeks	<0.007	<0.007	0.197	<0.211
4 weeks	<0.007	<0.007	0.084	<0.098
Bluegill Fillet Pond B				
1 hour	0.036	<0.007	0.006	<0.049
3 hours	0.022	<0.007	0.032	<0.061
6 hours	0.020	<0.007	0.109	<0.136
12 hours	<0.007	<0.007	0.207	<0.221
1 day	0.014	<0.023	0.286	<0.323
3 days	0.017	0.025	0.330	0.382
1 week	0.014	0.043	0.522	**0.589**
2 weeks	<0.007	0.019	0.298	<0.324
3 weeks	<0.007	<0.007	0.052	<0.064
4 weeks	<0.007	<0.007	0.043	<0.057

[a] Each value represents the average of 3-4 samples.

[b] Residues <LOD or <LOQ are added into totals as <0.007 and <0.023 ppm, respectively.

Table 23. Summary of average triclopyr and metabolite residues in **catfish fillet** from ponds treated to a target concentration of 2.5 ppm, from the **Missouri** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Catfish Fillet Pond A				
1 hour	0.016	<0.007	0.005	<0.028
3 hours	<0.023	<0.007	0.017	<0.049
6 hours	0.018	<0.007	0.194	<0.229
12 hours	<0.023	<0.007	0.787	<0.817
1 day	<0.023	0.023	1.367	<1.413
3 days	<0.023	0.023	1.169	<1.217
1 weeks	<0.023	0.023	1.992	<2.038
2 weeks	<0.007	0.023	2.062	**<2.092**
3 weeks	<0.007	<0.007	0.621	<0.635
4 weeks	<0.007	<0.007	0.246	<0.260
Catfish Fillet Pond B				
1 hour	0.025	<0.007	0.005	<0.037
3 hours	0.016	<0.007	0.118	<0.141
6 hours	<0.007	<0.007	0.116	<0.130
12 hours	<0.023	<0.007	0.645	<0.675
1 day	<0.023	0.023	1.184	<1.230
3 days	<0.007	0.023	1.734	<1.764
1 week	<0.007	0.023	2.941	**<2.971**
2 weeks	<0.007	0.012	1.226	<1.245
3 weeks	<0.007	<0.007	0.460	<0.474
4 weeks	<0.007	<0.007	0.206	<0.220

[a] Each value represents the average of 3-4 samples.

[b] Residues <LOD or <LOQ are added into totals as <0.007 and <0.023 ppm, respectively.

Table 24. Summary of average triclopyr and metabolite residues in **pond water**, treated to a target concentration of 2.5 ppm, from the **Texas** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total
Water Pond A				
1 hour	1.831	0.011	ND [b]	1.842
3 hours	2.384	0.014	ND	2.398
6 hours	2.310	0.013	0.002	2.325
12 hours	2.389	0.014	0.004	**2.407**
1 day	2.154	0.013	0.004	2.171
2 days	1.907	0.013	0.007	1.929
3 days	1.788	0.006	0.007	1.801
5 days	1.501	0.005	0.005	1.511
1 week	1.314	0.005	0.005	1.324
2 weeks	0.598	0.002	0.003	0.603
3 weeks	0.220	0.001	0.001	0.222
4 weeks	0.086	0.001	<0.001	<0.088
6 weeks	0.009	ND	<0.001	<0.010
12 weeks	ND	ND	ND	ND
Water Pond B				
1 hour	1.882	0.022	ND	1.904
3 hours	2.054	0.016	ND	2.070
6 hours	2.743	0.020	0.001	**2.764**
12 hours	2.361	0.013	0.004	2.378
1 day	2.153	0.020	0.004	2.177
2 days	2.263	0.012	0.007	2.282
3 days	1.993	0.010	0.005	2.008
5 days	1.624	0.008	0.004	1.636
1 week	1.332	0.007	0.005	1.344
2 weeks	0.566	0.002	0.003	0.571
3 weeks	0.192	0.001	0.002	0.195
4 weeks	0.070	<0.001	<0.001	<0.072
6 weeks	0.006	ND	ND	0.006
12 weeks	ND	ND	ND	ND

[a] Each value represents the average of 6-8 samples.

[b] ND = <0.0001 ppm; this figure was a not added to totals.

Table 25. Summary of average triclopyr and metabolite residues in **bluegill fillet** from ponds treated to a target concentration of 2.5 ppm, from the **Texas** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Bluegill Fillet Pond A				
1 hour	0.022	<0.007	0.006	<0.034
3 hours	0.025	<0.007	0.020	<0.052
6 hours	0.031	<0.023	0.124	<0.178
12 hours	0.029	0.014	0.510	<0.553
1 day	0.016	<0.023	0.260	<0.299
3 days	0.026	0.039	0.792	**0.857**
1 week	<0.023	<0.023	0.257	<0.303
2 weeks	<0.023	0.017	0.140	<0.180
3 weeks	<0.007	<0.007	0.074	<0.086
4 weeks	<0.007	<0.007	0.021	<0.035
Bluegill Fillet Pond B				
1 hour	0.029	<0.007	0.006	<0.042
3 hours	<0.023	<0.007	0.006	<0.036
6 hours	0.028	<0.007	0.074	<0.109
12 hours	0.047	<0.023	0.369	**<0.439**
1 day	0.027	<0.023	0.198	<0.248
3 days	0.019	0.026	0.149	0.194
1 week	<0.023	0.022	0.202	<0.247
2 weeks	<0.023	0.018	0.208	<0.249
3 weeks	<0.007	<0.023	0.099	<0.129
4 weeks	<0.007	<0.007	0.022	<0.036

[a] Each value represents the average of 3-4 samples.

[b] Residues <LOD or <LOQ are added into totals as <0.007 and <0.023 ppm, respectively.

Table 26. Summary of average triclopyr and metabolite residues in **catfish fillet** from ponds treated to a target concentration of 2.5 ppm, from the **Texas** site (MRID 44456103).

Sampling period	Residue concentration (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Catfish Fillet Pond A				
1 hour	<0.023	<0.007	0.017	<0.047
3 hours	0.016	<0.007	0.005	<0.028
6 hours	0.029	<0.007	0.070	<0.106
12 hours	0.033	<0.023	0.326	<0.382
1 day	0.017	<0.023	0.338	<0.378
3 days	0.017	<0.023	1.069	**<1.109**
1 week	<0.023	<0.023	0.451	<0.499
2 weeks	<0.023	<0.007	0.187	<0.217
3 weeks	<0.023	<0.007	0.145	<0.175
4 weeks	<0.007	<0.007	0.060	<0.074
Catfish Fillet Pond B				
1 hour	0.028	<0.007	<0.007	<0.042
3 hours	<0.023	<0.007	<0.007	<0.037
6 hours	0.028	<0.007	0.059	<0.094
12 hours	0.053	<0.007	0.629	**<0.689**
1 day	0.019	<0.023	0.433	<0.475
3 days	0.024	<0.023	0.536	<0.583
1 week	0.018	<0.023	0.519	<0.560
2 weeks	0.017	<0.007	0.204	<0.228
3 weeks	<0.023	<0.007	0.122	<0.152
4 weeks	<0.023	<0.007	0.064	<0.094

[a] Each value represents the average of 3-4 samples.

[b] Residues <LOD or <LOQ are added into totals as <0.007 and <0.023 ppm, respectively.

MRID 44456104. Texas pond study, 1996. Triclopyr aquatic testing was conducted at the USAE Waterways Experimental Station, Lewisville Aquatic Ecosystem Research Facility, near Lewisville, TX. A target water concentration of 2.5 ppm was achieved by applying two 9.5 L containers of the 3 lb ae/gal formulation via boat-mounted boom sprayer to a 2700 m^3 pond. Three water and sediment sampling stations were located to divide the pond in thirds. A second pond of 2920 m^3 with one sampling station was used to obtain control samples. The test pond was equipped with five cages for each of two test fish species, green sunfish (*Lepomis cyanellus*) and channel catfish (*Ictalurus punctatus*) placed at 20 fish per cage. The control pond contained two cages of 20 fish per species.

Water and fish were sampled twice daily on days 0-9, daily on days 10-18, every two days on from days 20-30, and every third day from days 30-42. Control samples were taken prior to the start of the test and on days 3 and 14. Water was refrigerated and analyzed using GC/MSD method GRM 95.18, within 2 weeks of sampling by Envirobiotech, Bernville, PA. The calculated LOD and LOQ for residues in water were 4×10^{-5} ppm and 1.45×10^{-4} ppm, respectively. Fish samples were refrigerated, then frozen for storage and analyzed by DowElanco, Indianapolis, IN using GC/MSD method GRM 97.02. The calculated LOD and LOQ for fish tissues were 0.005 ppm and 0.018 ppm, respectively. The maximum interval between fish sampling and completion of analyses was approximately 21 months.

Results of residue analysis are summarized in Tables 27 through 29. Maximum total residues in pond water were 2.439 ppm on Day 0 and consisted primarily of the parent compound; TCP and TMP concentrations were very low, ≤0.013 and ≤0.002 ppm, respectively. Total residues in water decreased to below 0.5 ppm on Day 13 after treatment. Residues in sunfish and catfish consisted almost entirely of TMP; beginning at the second sampling (Day 0, p.m.), TCP and TMP were <LOQ or <LOD in fillets of both species. Maximum total residues were <0.723 ppm in sunfish, reached on Day 3, and <2.036 ppm in catfish on Day 7. Residue concentration and composition were similar in sunfish viscera (data not shown) and fillets; TMP constituted most of the residue, with parent and TCP at <0.2 ppm. Residues were higher in catfish viscera (not shown), reaching >6.26 ppm on day 9 after treatment; parent and TCP remained at <0.08 and <0.04 ppm, respectively.

Table 27. Residues of triclopyr and metabolites in **water** from a pond in TX (1996) treated to a target concentration of 2.5 ppm (MRID 44456104).

Sampling day	Residues (ppm) [a]			
	Triclopyr	TCP	TMP	Total
Day 0, a.m..	2.426	0.013	ND [b]	**2.439**
Day 0, p.m.	2.087	0.004	NQ [b]	2.091
Day 1, a.m.	1.509	0.001	0.002	1.512
Day 1, p.m.	1.409	0.001	0.002	1.412
Day 2, a.m.	1.204	0.002	0.001	1.207
Day 2, p.m.	1.196	0.001	0.002	1.199
Day 3, a.m.	1.537	0.002	0.001	1.540
Day 3, p.m.	1.342	0.002	0.001	1.345
Day 4, a.m.	1.181	0.002	0.001	1.184
Day 4, p.m.	1.115	0.001	0.002	1.118
Day 5, a.m.	1.110	0.002	0.001	1.113
Day 5, p.m.	0.942	0.001	0.002	0.945
Day 6, a.m.	0.836	0.002	0.002	0.840
Day 6, p.m.	0.821	0.001	0.002	0.824
Day 7, a.m.	0.627	0.005	0.001	0.633
Day 7, p.m.	0.586	0.003	0.002	0.591
Day 8, a.m.	0.687	0.003	0.001	0.691
Day 8, p.m.	0.588	0.001	0.001	0.590
Day 9, a.m.	0.620	0.003	0.001	0.624
Day 9, p.m.	0.615	0.001	0.001	0.617
Day 10	0.569	0.004	0.001	0.574
Day 11	0.512	0.002	0.001	0.515
Day 12	0.544	0.003	0.001	0.548
Day 13	0.427	0.004	0.001	0.432
Day 14	0.378	0.006	0.001	0.385
Day 15	0.308	0.005	0.001	0.314
Day 16	0.267	0.003	0.001	0.271
Day 17	0.250	0.002	0.001	0.253
Day 18	0.274	0.001	0.001	0.276
Day 20	0.216	0.001	0.001	0.218
Day 22	0.145	0.001	0.001	0.147
Day 24	0.148	0.001	0.001	0.150
Day 26	0.085	0.001	<0.001	<0.087
Day 28	0.076	0.001	<0.001	<0.078
Day 30	0.054	0.001	<0.001	<0.056
Day 33	0.044	<0.001	<0.001	<0.046
Day 36	0.027	<0.001	<0.001	<0.029
Day 39	0.020	<0.001	NQ	<0.021
Day 42	0.013	<0.001	NQ	<0.014

[a] Each value represents the average of three samples; data were corrected for recovery by petitioner.

[b] ND = 4×10^{-5} ppm. NQ = 1.45×10^{-4} ppm. These values were not included in the total residue calculations.

Table 28. Residues of triclopyr and metabolites in **green sunfish** fillets from a pond in TX (1996) treated to a target concentration of 2.5 ppm (MRID 44456104).

Sampling day	Residues (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Day 0 , a.m.	<0.018	<0.005	<0.005	<0.028
Day 0 , p.m.	<0.018	<0.018	0.2	<0.236
Day 1 , a.m.	<0.018	<0.005	0.1	<0.123
Day 1 , p.m.	<0.018	<0.018	0.3	<0.339
Day 2, a.m.	<0.005	<0.018	0.1	<0.123
Day 2, p.m.	<0.018	<0.018	0.4	<0.436
Day 3, a.m.	<0.005	<0.018	0.2	<0.223
Day 3, p.m.	<0.005	<0.018	0.7	**<0.723**
Day 4, a.m.	<0.018	<0.005	0.1	<0.123
Day 4, p.m.	<0.005	<0.018	0.4	<0.423
Day 5, a.m.	<0.005	<0.018	0.1	<0.123
Day 5, p.m.	<0.005	<0.018	0.1	<0.123
Day 6, a.m.	<0.005	<0.018	0.2	<0.223
Day 6, p.m.	<0.005	<0.018	0.3	<0.323
Day 7, a.m.	<0.005	<0.018	0.4	<0.423
Day 7, p.m.	<0.018	<0.018	0.2	<0.236
Day 8, a.m.	<0.005	<0.005	0.1	<0.111
Day 8, p.m.	<0.005	<0.018	0.3	<0.323
Day 9, a.m.	<0.005	<0.018	0.2	<0.223
Day 9, p.m.	<0.005	<0.018	0.2	<0.223
Day 10	<0.005	<0.005	0.1	<0.110
Day 11	<0.005	<0.018	0.4	<0.423
Day 12	<0.005	<0.018	0.3	<0.323
Day 13	<0.005	<0.018	0.2	<0.223
Day 14	<0.005	<0.005	0.1	<0.110
Day 15	<0.005	<0.018	0.1	<0.123
Day 16	<0.005	<0.018	0.1	<0.123
Day 17	<0.005	<0.005	0.4	<0.410
Day 18	<0.005	<0.018	0.2	<0.223
Day 20	<0.005	<0.018	0.1	<0.123
Day 22	<0.005	<0.005	0.5	<0.510
Day 24	<0.005	<0.005	0.2	<0.210
Day 26	<0.005	<0.005	0.3	<0.310
Day 28	<0.005	<0.005	0.1	<0.110
Day 30	<0.005	<0.005	0.1	<0.110
Day 33	<0.005	<0.005	<0.005	<0.015
Day 36	<0.005	<0.005	<0.018	<0.028
Day 39	<0.005	<0.005	<0.005	<0.015
Day 42	<0.005	<0.005	<0.005	<0.015

[a] Each value represents one sample; data were corrected for recovery by petitioner.
[b] Residues <LOD or <LOQ are added into totals as <0.005 and <0.018 ppm, respectively.

Table 29. Residues of triclopyr and metabolites in **catfish** fillets from a pond in TX (1996) treated to a target concentration of 2.5 ppm (MRID 44456104).

Sampling Day	Residues (ppm) [a]			
	Triclopyr	TCP	TMP	Total [b]
Day 0, a.m.	<0.018	<0.018	0.1	<0.136
Day 0, p.m.	<0.018	<0.018	0.7	<0.736
Day 1, a.m.	<0.018	<0.018	1.2	<1.236
Day 1, p.m.	<0.018	<0.018	<0.005	<0.041
Day 2, a.m.	<0.005	<0.018	0.7	<0.723
Day 2, p.m.	<0.005	<0.018	1.1	<1.123
Day 3, p.m.	<0.018	<0.018	0.8	<0.844
Day 4, a.m.	<0.005	<0.005	0.6	<0.610
Day 4, p.m.	<0.018	<0.018	1.2	<1.236
Day 5, a.m.	<0.005	<0.018	0.9	<0.923
Day 5, p.m.	<0.018	<0.018	0.6	<0.636
Day 6, a.m.	<0.005	<0.018	0.9	<0.923
Day 6, p.m.	<0.018	<0.018	1.2	<1.236
Day 7, p.m.	<0.018	<0.018	2.0	<2.036
Day 8, a.m.	<0.005	<0.018	1.3	<1.323
Day 8, p.m.	<0.005	<0.018	0.8	<0.823
Day 9, a.m.	<0.005	<0.018	1.6	<1.623
Day 9, p.m.	<0.005	<0.018	1.4	<1.423
Day 10, a.m.	<0.005	<0.018	1.0	<1.023
Day 11	<0.005	<0.018	0.4	<0.423
Day 12	<0.005	<0.018	0.7	<0.723
Day 13	<0.005	<0.018	0.3	<0.323
Day 14	<0.005	<0.018	0.4	<0.423
Day 15	<0.005	<0.018	1.0	<1.023
Day 16	<0.005	<0.018	0.4	<0.423
Day 17	<0.005	<0.018	0.5	<0.523
Day 18	<0.005	<0.005	0.1	<0.110
Day 20	<0.005	<0.005	0.4	<0.410
Day 22	<0.005	<0.005	0.2	<0.210
Day 24	<0.005	<0.005	0.2	<0.210
Day 26	<0.005	<0.005	0.2	<0.210
Day 28	<0.005	<0.005	0.1	<0.110
Day 30	<0.005	<0.005	0.1	<0.110
Day 33	<0.005	<0.005	<0.005	<0.015
Day 36	<0.005	<0.005	0.1	<0.110
Day 39	<0.005	<0.005	<0.018	<0.028
Day 42	<0.005	<0.005	<0.018	<0.028

[a] Each value represents one sample; data were corrected for recovery by petitioner.

[b] Residues <LOD or <LOQ are added into totals as <0.005 and <0.018 ppm, respectively.

Conclusions

Current HED policy recommends regulating residues found in the *edible portions* of fish and shellfish. In the submitted studies, the maximum combined residue in fish edible portions was 2.97 ppm. The maximum combined residues in shellfish edible portions were 0.523 ppm in clams and 0.454 ppm in crayfish. In a study previously reviewed ("91-MN-06. Proposed Section 18 exemption for the use of triclopyr in or near aquatic sites", D. McNeilly, 03-APR-1991), maximum combined residues were 3.44 ppm in clams (edible portion) and 4.87 ppm in crayfish (whole organism including shell) from Lake Seminole, GA treated to a concentration of 2.5 ppm triclopyr in the water. Because the crayfish were analyzed as whole organisms, it is not appropriate to use these data to set tolerance levels. Consequently, the appropriate tolerance levels for combined residues of triclopyr, TCP, and TMP in fish and shellfish are 3.0 and 3.5 ppm, respectively. **A revised Section F specifying these tolerance levels should be submitted.**

In water, total residues of triclopyr, TCP, and TMP, were <0.5 ppm after 3 weeks in all cases, with the exception of one pond site in CA, where residues were 0.645 ppm at 3 weeks and <0.5 ppm after 4 weeks. Residues were <0.5 ppm at a set-back distance of 100 m from the treated plot.

Anticipated Residues

For the purposes of anticipated residues for use in a dietary exposure analysis, Table 30 summarizes the maximum combined residues of triclopyr, TCP, and TMP measured in the submitted studies.

Table 30. Anticipated Residues of Triclopyr, TCP, and TMP in Fish and Shellfish for use in Dietary Exposure Analysis of Triclopyr[a].

Maximum Combined Residues of Triclopyr, TCP, and TMP in Fish (ppm)	Maximum Combined Residues of Triclopyr, TCP, and TMP in Shellfish (ppm)
0.952	3.44[b]
0.670	0.405
0.455	0.523
0.754	0.310
0.275	0.454
0.463	
0.678	
0.954	
0.887	
0.569	
0.956	

Maximum Combined Residues of Triclopyr, TCP, and TMP in Fish (ppm)	Maximum Combined Residues of Triclopyr, TCP, and TMP in Shellfish (ppm)
0.695	
0.589	
2.092	
2.971	
0.857	
0.439	
1.109	
0.689	
0.723	
2.036	
0.943 (Mean value)	1.03 (Mean value)

[a] Except where indicated otherwise, all residue values taken from studies reviewed in this memo.

[b] Maximum value from previous review: ("91-MN-06. Proposed Section 18 exemption for the use of triclopyr in or near aquatic sites", D. McNeilly, 03-APR-1991).

OPPTS GLN 860.1480: Meat, Milk, Poultry, Eggs

The proposed tolerances would not alter the maximum theoretical dietary burdens (MTDBs) of triclopyr for cattle or poultry estimated in conjunction with the existing feeding studies for grass and rice. Additional feeding study data are not required.

OPPTS GLN 860.1850/1900: Confined/Field Accumulation in Rotational Crops

There are no rotational crop issues associated with the proposed use.

Codex Issues

There are no established or proposed Codex, Canadian, or Mexican MRLs for triclopyr residues (see Attachment 2). Therefore, harmonization is not an issue at this time.

Agency Memoranda Cited in this Review

CBTS Nos.	8078, 8079, 8223, 8224, 8561, 8562, 8786, 8793, and 8962-8964
Subject:	PP#1F0394 and PP#1F03935. Triclopyr DowElanco Rice Herbicide on Rice Grain and Straw and Triclopyr Aquatic Herbicide on Various Aquatic Sites.
From:	G. Otakie
To:	R.J. Taylor/E.J. Allen, RD
Date:	9/4/92
MRIDs:	41200304, 41714304-07, 41865901

DP Barcode:	D210628
Subject:	Triclopyr Aquatic Herbicide - Evaluation of Field Study Protocol.
From:	G. Otakie
To:	R.J. Taylor/E.J. Allen, RD
Date:	4/20/95
MRID:	None

DP Barcode:	D225012
Subject:	Triclopyr. Product and Residue Chemistry Chapters for the Reregistration Eligibility Decision Document (RED).
From:	W. Smith
To:	P. Deschemp, RCAB
Date:	7/23/96
MRID:	None

DP Barcode:	D264679
Subject:	PP#1F3935. Triclopyr in/on Fish, Shellfish, and Drinking Water. Request for Petition Method Validation (PMV)
From:	W. Donovan, RAB1
To:	F.D. Griffith, ACB
Date:	4/7/00
MRID:	44456108 and -12

ATTACHMENT 2

INTERNATIONAL RESIDUE LIMIT STATUS

Chemical Name: (3,5,6-trichloro-2-pyridinyl)oxyacetic acid	Common Name: Triclopyr	X□ Proposed tolerance □ Reevaluated tolerance □ Other	Date: 29-MAR-2001

Codex Status (Maximum Residue Limits)	U. S. Tolerances
X No Codex proposal step 6 or above □ No Codex proposal step 6 or above for the crops requested	Petition Number: 1F03935 DP Barcode: D268064 Other Identifier:
Residue definition (step 8/CXL): N/A	Reviewer/Branch: W. Donovan/RAB1
	Residue definition: Triclopyr, TCP, and TMP

Crop (s)	MRL (mg/kg)	Crop(s)	Tolerance (ppm)
		Fish	3.0
		Shellfish	5.0
		Water (requested allowable water residue level)	0.5

Limits for Canada	Limits for Mexico
X No Limits □ No Limits for the crops requested	X No Limits □ No Limits for the crops requested
Residue definition: N/A	Residue definition: N/A

Crop(s)	MRL (mg/kg)	Crop(s)	MRL (mg/kg)

Notes/Special Instructions: S. Funk, 04/30/01

Rev. 1998